Chapter 1

Introduction

One of the main tasks of modern materials science is the tailoring of new materials with the properties desired. This tailoring is strongly connected with the characterization of structure, composition, morphology and the structure-property relationships at nanoscale. An important aspect of structure characterization is the direct imaging of materials. Electron microscopy is a powerful method for imaging, diffraction and spectroscopy among the various analytical techniques using different wavelengths and instruments.

Since the first transmission electron microscope (TEM) was built by Max Knoll and Ernst Ruska in 1932, TEM has been proved as one of the most important microscopy tools for the investigation of the structure and composition of materials down to the atomic scale. Compared with other microanalysis methods such as scanning probe microscopy (SPM) or X-ray diffraction, TEM has several significant advantages. First, the high energy electrons accelerated in a TEM (80-1000 keV) have an extreme short wavelength and push the diffraction limitation down to picometer scale. Then more fine spatial structures can be resolved by TEM. In the state of the art instruments, sub-angstrom resolution has been achieved by installing spherical aberration (Cs) correctors for the objective lens and the electron probe. Second, the high energy electrons transmit through the whole thin specimen and carry the bulk information, while conventional SPM is only sensitive to the surface structure. Third, compared with X-rays, electrons can be focused much easier, thus scattered electron waves can be detected in both the reciprocal space (diffraction mode) and the real space (imaging mode). Additionally, the focused electrons can be used to probe the local structure and composition down to atomic scale, while the signal in the X-ray diffraction is normally coming from a larger area at micrometer scale. Finally, based on the different interactions between the incident electrons and the thin specimen (elastic and inelastic, or coherent and incoherent scattering), various TEM imaging and spectroscopy techniques such as energy dispersive X-ray spectroscopy (EDXS), electron energy loss spectroscopy (EELS) and scanning transmission electron microscopy (STEM) have been developed for the characterization of structure and composition.

The interaction between the incident electrons and the specimen is an electron scattering process by the object potential, including the electrostatic potential and the magnetic vector potential. The electrostatic potential is generated by the positive charges of the nucleus and the negative electron charges in the material and connected with the charge density distribution via Poison equation. It is well known that the charge density distribution including the atom positions, the electronic structure and the interatomic bonding dominates the fundamental properties of the materials. Similarly, the distribution of magnetic vector potential depends on the specimen magnetization and is related to the magnetic properties of the material. Thus, from a general point of view, one of the main tasks of TEM is detecting the scattered electron waves to reconstruct the scattering potential, and further to achieve the information about the related structure, morphology, composition and particular properties.

However there is a well known problem in conventional TEM, that only the amplitude (intensity) of the scattered electron wave, but not the phase can be recorded. The missing phase information limits the further applications of TEM, particularly for the so-called phase objects. Those objects only cause a slight or even no change of scattered electron amplitudes, but a more significant change of the electron phases. Thus, important information is lost with the phase.

In 1948, Dennis Gabor firstly proposed the holography method for solving the phase problem. As he suggested, by using a known reference wave to superimpose with the scattered electron wave, the full amplitude and phase information will be recorded in the interference fringe image, called electron hologram. After the reconstruction, the amplitude and phase information can be successfully separated.

Today electron holography has been widely used in both high resolution and medium resolution TEM applications. In high resolution electron holography, electron phase shifts are imaged at atomic scales and reflect the rapid varied atomic potential information. Thus, high resolution electron holography can help to get a thorough understanding of the atomic structure of the materials to be investigated.

Furthermore, there is a great interest in medium resolution electron holography. At medium resolution, the electron phase shift induced by a more slowly varying electrostatic and magnetic potential will be imaged and reflect the distributions of the electromagnetic field at nanoscale. These slowly varied potentials include the electrostatic potential generated by the inner field of p-n junctions, ferroelectric domains or the mean inner potential of conventional materials, and the magnetic vector potential of magnetic materials. Applying high energy electrons, these slowly varied scattering potentials (compared with atomic scale) act more like a weak or pure phase object. Then they are difficult to be imaged in a conventional amplitude (intensity) image but show more significant contrast in the phase image. Then quantitative information of the electromagnetic field distributions at nanoscale can be further induced from the phase image. This is particular interesting for magnetic, semiconducting and ferroelectric materials research, since the phase imaging method extends the applications of commonly used TEM from the characterization of structure and composition to the analysis of structure-property relationships.

This work mostly focuses on the applications of electron holography at medium resolution. In the first topic of this thesis, we will describe electron holography and Lorentz microscopy for the observation of the magnetic domain structure and the correlations between microstructure and magnetic domain structures in FeCo-based and Fe-based nanocrystalline soft magnetic alloys. In the second topic, we will deal with 3D-(Si,Ge) semiconductor nanostructures investigated by a 2D-phase mapping. The content of the thesis is divided into the following parts:

Following this introduction (chapter 1), an introduction to the theoretical and practical methods of electron holography, including the hologram formation, recording and phase reconstruction is given in chapter 2. The main parameters and the influence of the instrument performance on the phase image will also be discussed.

In chapter 3, the investigation of the microstructure of FeCo-based nanocrystalline alloys by advanced TEM techniques is discussed carefully, including diffraction contrast imaging, electron diffraction, HRTEM, STEM-HAADF as well as EDXS.

The study of magnetic domain structure and the correlations between microstructure and magnetic domain structure in FeCo-based and Fe-based nanocrystalline alloys will be treated in chapter 4. First the magnetic contrast formation in electron holography will be discussed in detail. Then, the observation of magnetic domain structures by means of Lorentz microscopy and electron holography will be illustrated. The domain wall width was measured and compared with a numerical simulation. The quantitative magnetic flux density was measured by a special procedure, where EELS measurements for the determination of thickness were utilized. Furthermore, the dynamical magnetization was observed in both the Lorentz microscopy and electron holography. The correlations between the microstructure and the magnetic domain structure will be discussed. The properties of the magnetic domain structure of a new type of B free Fe-based nanocrystalline alloys will also be presented.

Chapter 5 deals with the 3D-imaging of (Si,Ge) islands from a 2D-phase mapping. First, the basic principle is described for 3D-reconstruction of the nanostructure from 2D-phase mapping. The relationship between specimen thickness and electron phase shift due to the mean inner potential will be discussed. Then the experimental phase mapping and digital phase processing are presented. Following two different models, viz., the isolated atom model and the “bonded atom” model, the mean inner potential of (Si,Ge) alloys is calculated and applied for the 3D reconstruction.